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# METHANE AND HYDROGEN GAS SENSING PROPERTIES OF FULLERENES C60 FOR BREATH ANALYZING PURPOSES

BY

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**Abstract.** Hydrogen and methane breath tests (BTs) have been used successfully to detect gastrointestinal disorders, for diagnosis of carbohydrate maldigestion syndromes and small intestinal bacterial overgrowth (SIBO). BT is a useful, inexpensive, simple and safe test in the diagnosis of carbohydrate maldigestion, methane-associated constipation, and evaluation of bloating/gas disorders. Carbon nanomaterials are very promising for the design of new sensors capable to detect breath compounds and recently a steady growth of the research efforts and published papers on the use of such materials as sensitive element was noticed. Multi-layer fullerene-C60, a very promising member of carbon nanostructure family, has been the object of extensive research since its discovery due to the presence of its synthesis-related structural defects, a highly required characteristic in the development of room temperature sensing devices. In this work we present a chemiresistor gas sensor (CGS) based on fullerenes operating at room temperature, displaying an enhanced sensitivity to $H_2$ and $CH_4$.

**Key words:** fullerenes; breath test; dielectrophoresis; sensitivity; chemirestive gas sensor.

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## 1. Introduction

The use of nanotechnology-derived products in the development of sensors and analytical measurement methodologies has increased significantly over the past decade. Nano-based sensing approaches supposed the use of nanoparticles (NPs) and nanostructures to enhance sensitivity and selectivity, improve sample preparation and increase portability. Breath testing is one research area that benefits from the development of such sensors (Konvalina & Haick, 2016; Goodarzi *et al*. 2017; Tiwari *et al*., 2016; Waud *et al*., 2008). BT is useful in the diagnosis of carbohydrate maldigestion, methane-associated constipation, and evaluation of bloating/gas. A rise in hydrogen of ≥ 20 p.p.m. by 90 min during glucose or lactulose BT for SIBO is considered positive while methane levels above 10 p.p.m. is considered methane-positive. Also a rise in hydrogen of over 20 p.p.m. from baseline during BT is considered positive for maldigestion (Rezaie *et al*., 2017). Current trends in the development of low-cost portable NP-based technology for rapid assessment of breath analyzer as well as challenges for practical implementation and future research were focused towards the use of carbon nanoparticle family. Room temperature operation, low detection limit and fast response time are highly desirable for a wide range of gas sensing applications. However, the common available gas sensors suffer mainly from high temperature operation or external stimulation for response and recovery.

Researchers have developed chemiresitor-type sensors based on NP aimed to detect gases down to parts-per-million (ppm) (Peng *et al*., 2009; Donato 2011 *et al*.; Deb and Chusuei, 2011; Khan *et al*., 2016; Dasari *et al*., 2017; Wang & Yeow, 2009). These sensors are capable to operate at room temperature without the need of an external heating source, by interacting with gas molecules and, depending on reducing or oxidizing properties inject or extract electrons from NPs providing an electrical signal that can be measured between the electrode's terminals (Cui *et al*., 2012; Shobin & Manivannan, 2014). Thus, these sensors can be integrated into smart portable devices to make BT measurements necessary to detect the gut related diseases. Part of the carbon fullerene family, onion-like carbons (OLCs) or carbon nano-onions (CNOs) are small, spherical, zero-dimensional (0-D) structures of 5 nm to 10 nm consisting of concentric shells of graphitic carbon (Bartelmess *et al*., 2014). These nanoparticles are being synthesized through electron-beam irradiation, arc discharge between two graphite electrodes in water, vacuum annealing of nanodiamond (ND) powder at 1,800°C or laser ablation (Fig. 1).

The OLCs structures used in our study have been obtained through annealing of carbon nanodiamonds in vacuum or in controlled atmosphere at slightly positive pressure in the presence of Helium (He) or Argon (Ar). This process leads to a productivity of over 90% and a higher purity of the nanodiamond. Nanodiamond produced through this method had a 5,...,7 nm diameter and OLCs with 6 to 8 layers. Spherical OLCs where obtained through

detonation of nanodiamond at 1,650°C. The detailed preparation procedure of OLCs is presented in (Plonska-Brzezinska *et al.*, 2011).

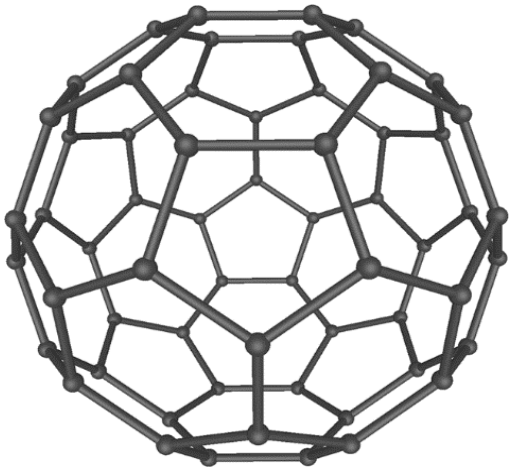


Fig. 1 − Representation of Fullerenes C60.

Romanenko has investigated application of these fullerenes in (Romanenko *et al.*, 2008), the author evaluated the influence of temperature on the conductivity of onion-like carbon (OLC) under exposure to various gas environments. Breczko investigated the OLCs as biosensing device within (Breczko *et al.*, 2012). He presented the development of carbon nano-onion (CNO) and poly(diallyldimethylammonium chloride) (PDDA) composite for detection of dopamine in the presence of ascorbic (AA) and uric (UA) acids. Joanna Luszczyn and her colleges reported in (Luszczyn *et al.*, 2010) the first covalent functionalization of oxidized CNOs (ox-CNOs) with biomolecules, and the tests showed excellent cytocompatibility of all CNOs, so these carbon nanostructures can be safely used for biological applications. (Bobrowska *et al.*, 2017) combines the hydrophilicity of surfactants with the robustness of carbon structures to produce carbon nano-onion/surfactant (CNO/surfactant) composites with superior and unusual physicochemical properties to assay the biological activity of well-dispersed CNO/surfactant composites against a strain of Escherichia coli. (Yang *et al.*, 2016) studies the electrochemical performance of carbon nano-onions derived from nanodiamonds and discovers the remarkable electrochemical activities of CNOs with high sensitivity, high selectivity and stable electrode responses for the detection of biologically important molecules in comparison to the multi-wall carbon nanotubes (MWCNTs), graphene nanoflakes GNFs and glassy carbon (GC).

The OLCs have some advantages for developing the sensitive element of the sensor due to their small dimensions as the distances between the carbon layers are only 0.34 nm, enhanced solubility, improved dispersion in the solvent

and better dispersion stability (Ugarte, 1992), a high temperature stability (Reinert *et al*., 2015), large aspect ratio, and non-porous texture. Although various researchers have developed sensing application for functionalized CNOs, a chemiresistor based on pristine nanoparticle as sensing probe for gaseous substances such as methane and hydrogen is highly requested, as it would simplify the procedure and manufacturing time. Our results demonstrate the proof-of-concept for a chemiresistive gas sensor (CGS) to be used in several applications including breath analyzers for hydrogen and methane. The authors demonstrated that the OLCs based sensor can detect $H_2$ and $CH_4$ at 10 ppm at room temperature in a minute time.

## 2. Sensor Manufacturing

The CGSs were fabricated by dielectrophoresis (DEP). The principle of DEP relies on the fact that a polarizable particle suspended in a fluid can be displaced under a nonuniform electric field. The particle will be displaced towards or away from areas of stronger field when its permittivity is higher or lower than that of the suspending fluid (Olariu and Arcire, 2017). OLCs were electromanipulated onto the gold interdigitated microelectrodes patterned on glass substrate (L 22.8 mm × W 7.6 mm × H 0.7 mm) provided by DropSens, Spain. The microelectrode, manufactured by photolithographic techniques, is composed of two interdigitated electrodes with two connection tracks, all made of gold with 250 × 2 digits and 6,760 µm digit length. With 5-µm gap between the digits and 5-µm width of the digits ensures a satisfactory distribution of electric field gradient.

In the case of a spherical particle (Fig. 1), the dielectrophoretic (DEP) force can be expressed as:

$$F_{\mathrm{DEP}} = 2\pi r^3 \varepsilon_0 \varepsilon_m \Re\mathrm{e}[K(\omega)] \nabla E^2, \tag{1}$$

where: $r$ is the radius of the spherical particle, $\varepsilon_0$ – the permittivity of free space, $\varepsilon_m$ – the permittivity of the fluid, $E$ – the local electric field. The $\Re\mathrm{e}[K(\omega)]$ parameter is the real part of Clausius-Mossotti factor:

$$K(\omega) = \frac{\varepsilon_p^* - \varepsilon_m^*}{\varepsilon_p^* + 2\varepsilon_m^*}, \tag{2}$$

where: $\varepsilon_0^*$ and $\varepsilon_m^*$ are the complex permittivities of the suspended particle and the fluid, respectively. Practically, depending on $\Re\mathrm{e}[K(\omega)]$ polarization the exerted DEP force will be positive or negative. If the particle's permittivity would be higher than the fluid's permittivity, a positive DEP force will be exerted, while in the other case a negative DEP force will be experimented (Fig. 2).

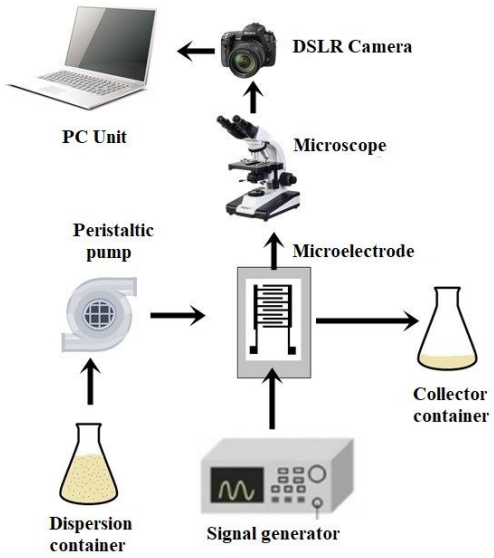
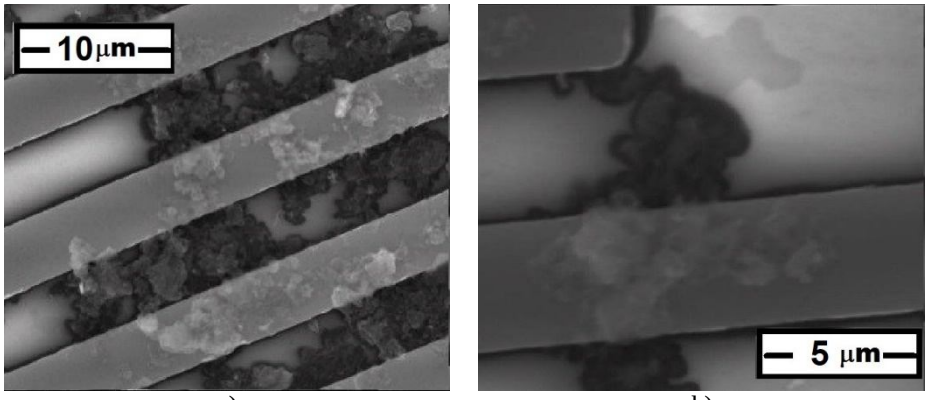



Fig. 2 − Schematic view regarding of the trapping process of the OLCs between the microelectrode's fingers.

To obtain the desired dispersion of OLCs in ethanol, a solution with 0.1µg/1ml of OLCs/ethanol concentration was made, firstly being stirred for one hour to break the large agglomeration of OLCs and then the dispersion was ultrasonicated for two hours in a 130 W ultrasonic bath at 28 kHz frequency. Particle trapping on microelectrode was accomplished by setting an arrangement setup based on a signal generator, a power amplifier to feed to the microelectrode and an acrylic chamber filled with a freshly developed dispersion of OLCs/ethanol through a peristaltic pump where the microelectrode was immersed.

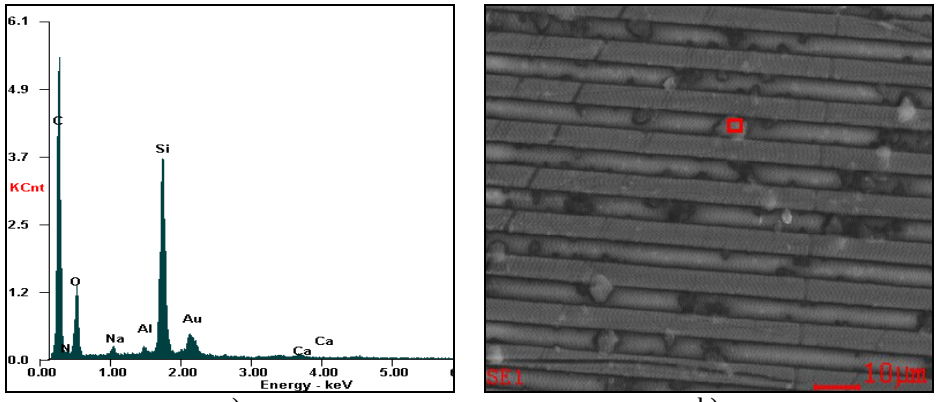
All CGSs were manufactured by immersing the electrodes in the dielectrophoretic acrylic chamber, holding the signal amplitude at 20Vpp while frequencies were varied at a) 50 kHz b) 100 kHz and c) 150 kHz. Another set of sensors were made by maintaining the frequency at 100 kHz, and varying the signal amplitude to 10 and 30 Vpp, respectively. Finally, the resistance of all the four sensors were measured with a digital multimeter. Particle trapping at the level of microelectrodes was also confirmed by scanning electron microscope (SEM) and Energy Dispersive X-Ray Analysis (EDX) as seen in Fig. 3 a) and b).

An EDX analysis is presented in Fig. 4 a) and b) an SEM image with the analysis spot. The presence of carbon is ascribed to the OLCs trapped between the electrodes, Si and O to the structure of glass but also it is noticed the presence of some other particles due to contamination with impurities bonded on structural defects of the OLCs.




a) b)

Fig. 3 – SEM image based on Quanta 200 (FEI) microscope, operating at 20 kV with secondary electrons in Low vacuum mode (LFD detector) of a) the OLCs clusters bridging the gap between the electrodes and b) detailed view between two electrodes.




a) b)

Fig. 4 − a) EDX analysis plot view of OLCs showing the presence impurities and b) SEM image with the location spot of EDX analysis.

Raman spectra for the trapped OLCs is plotted in Fig. 5. Raman spectroscopy was used as method to study the defect state of the OLCs. The presence of the two higher peaks, the D and the G band are a Raman spectrum signature of graphitic sp2 materials. The presence of randomly distributed impurities or surface charges in the OLCs, determines the G-peak to split into two peaks, G-peak and D-peak, the localized vibrational modes of the impurities interact with the extended phonon modes of OLCs leading to observed splitting. The first peak arises at around 1,350 $cm^{-1}$, the D-mode, generated by the presence of defects in the OLC structure. The presence of

disorder in sp2-hybridized carbon systems results in resonance Raman spectra. At 1,583 $cm^{-1}$, the G-mode is detected; the presence of this peak can be attributed to the tangential vibrations of carbon atoms with sp2-hybridization and is used to describe their crystalline state. The G-mode is present due to E2g mode at the Γ-point. The G-band arises from the stretching of the C-C bond in graphitic materials and is common to all sp2 carbon systems. The 2D-mode is observed at around 2,633 $cm^{-1}$, and it is corresponding to an overtone of the defect-related D-mode, its intensity being proportional to the sample purity level. The D+G band is also a defect-activated process as the D-band is.

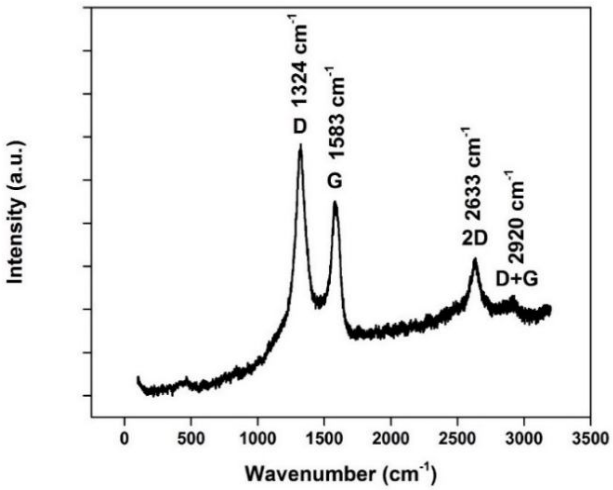



Fig. 5 − Raman spectra of OLCs.

In addition, electrical characterization was performed through current-voltage (I-V) characterization of all sensors with a Keithley 2635 source meter interfaced with a personal computer (PC) by (General Purpose Interface Bus) GPIB interface, which confirms the "ohmic" contacts forming between the microelectrode's fingers. The I-V characteristics for the four sensors: S1(50 kHz 20Vpp), S2(100 kHz 20Vpp), S3(150 kHz 20Vpp), S4(100 kHz 10Vpp), S5(100 kHz 30Vpp) are presented in Fig. 6. The measurements were taken at 25C°, with 50% humidity. All characteristics were fitted with a linear function within Originlab OriginPro 2016:

$$Y1 = 0.02018 \times X - 4.56171 \times 10^{-4},\ R2 = 0.99973, \quad (3)$$

$$Y2 = 0.0252 \times X - 3.15864 \times 10^{-4},\ R2 = 0.99956, \quad (4)$$

$$Y3 = 0.01685 \times X - 1.52593 \times 10^{-4},\ R2 = 0.99989, \quad (5)$$

$$Y4 = 0.01419 \times X - 1.91772 \times 10^{-4},\ R2 = 0.99987, \quad (6)$$

$$Y5 = 0.00752 \times X - 1.2381 \times 10^{-6},\ R2 = 0.99999. \quad (7)$$

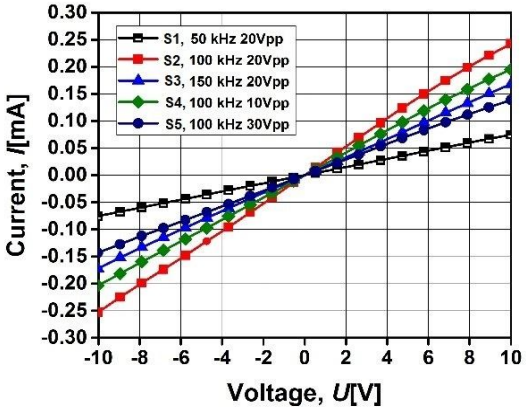



Fig. 6 − I-V characteristics for all sensors: S1 (50 kHz 20Vpp) , S2 (100 kHz 20Vpp), S3(150 kHz 20Vp), S4 (100 kHz 10Vpp), S5 (100 kHz 30Vpp).

## 3. Sensor Testing

The influence of the targeted gases on the manufactured sensors' resistance was studied through an experimental setup based on the schematic view plotted in Fig. 7.

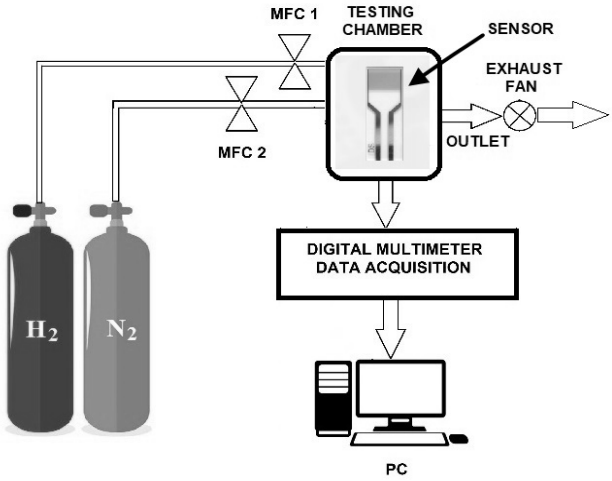
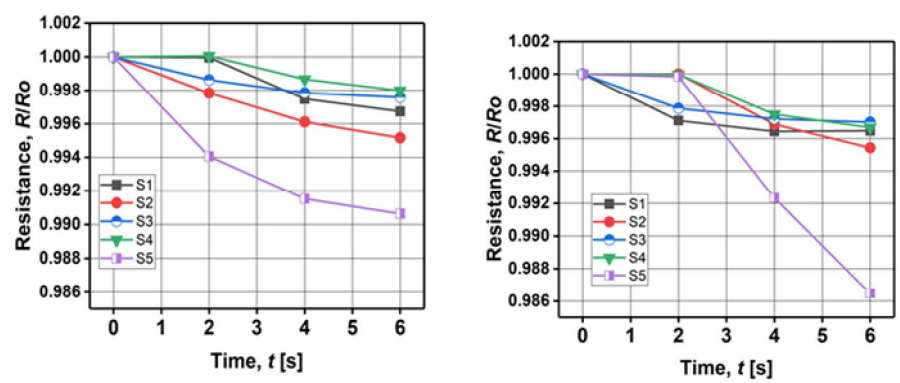



Fig. 7 − Experimental setup.

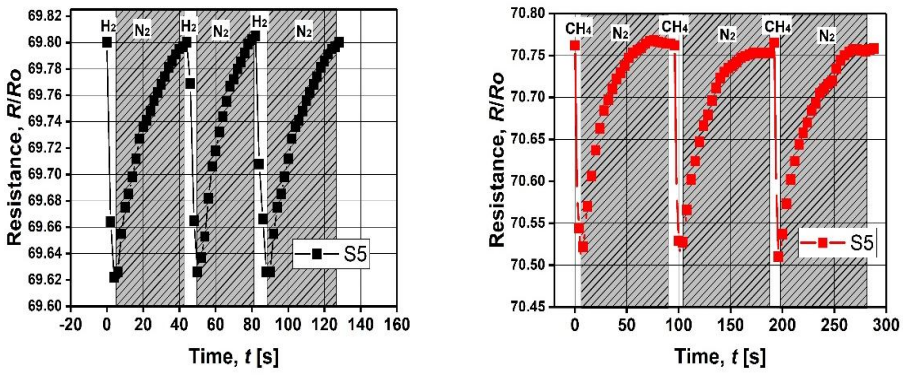
All CGSs were exposed to $H_2$ and $CH_4$, respectively, by alternating exposure to the analyte gas (10 ppm) and $N_2$ (Fig. 8 a and b). The normalized resistance values response shows that sensor S5 exhibits a pronounced reaction to $H_2$ and $CH_4$. The functionality of the sensor is proven in the plots from Fig. 9

under repeated exposure to $H_2$ and $CH_4$. The S5 sensor has a good response time, until complete desorption of the targeted analyte, under 40 s for $H_2$ and 80s for $CH_4$. The S2 and S5 sensors exhibit the most sensitive response to $H_2$ and $CH_4$, fact we can ascribe to the manufacturing procedure as both sensors were fabricated by employing a 100 kHz frequency. The employed frequency was experimentally determined in our earlier paper (Olariu and Arcire, 2017). At this frequency and at voltages of 20Vpp and 30 Vpp a higher amount of OLCs is trapped at the level of interdigitated microelectrode (IDE). This aspect plays a key role in an even distribution of the ethanol suspended nanoparticles on the surface of the IDE.




a) b)

Fig. 8 − Normalized resistance change response time to a) $H_2$ and b) $CH_4$.




a) b)

Fig. 9 − Repeated exposures of S5 to a) $H_2$ and b) $CH_4$.

A comparison study was undertaken to determine the influence of both gas analytes on each sensor's resistance change (normalized values). On the response slope, all sensors tend to exhibit a faster response to methane than to hydrogen (Fig. 10).

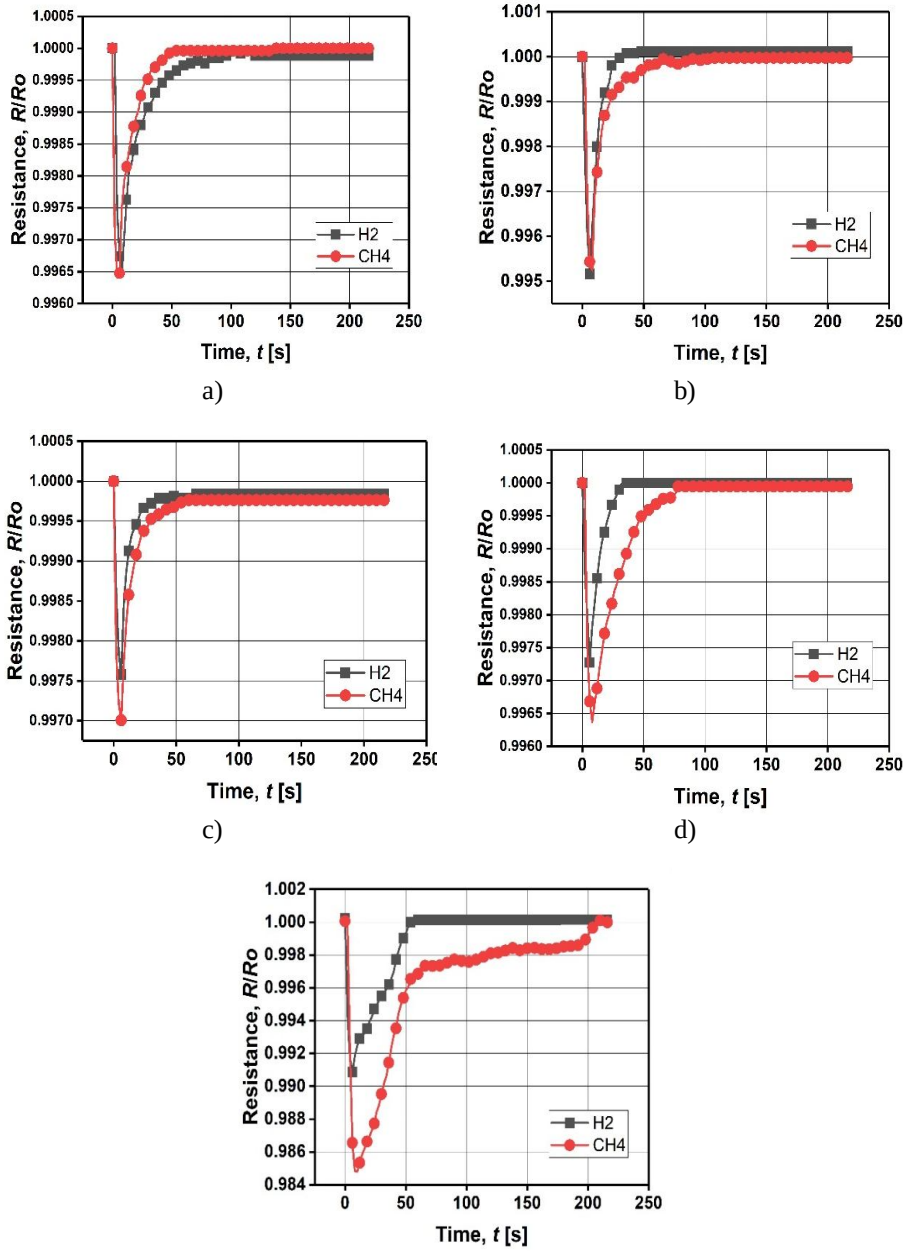



Fig. 10 − Response/Recovery time a) S1, b) S2, c) S3, d) S4, e) S5.

With the exception of sensor S1, the desorption time curve tends to have a slightly different allure, $CH_4$ molecules having a tendency to desorb

slower (unbound slowly). Resistance change is directly linked to the number of molecules bonded on the OLCs defects, thus a higher number of molecules require a longer time to desorb naturally at room temperature without an external stimulus such as an external heat source. All sensors achieve complete desorption within 60 seconds under exposure to $H_2$, and over 200s under exposure to $CH_4$ of sensor S5.

The fullerene C60 nanoparticles exposed to ethanol, during ultrasonication, can present several solvation shells around the central fullerene molecule. In the first layer of the solvation shell, ethanol molecules tended to form non-polar alkyl groups exposed to the surface, which contributes to the stimulation of structural defects of the OLC layers. The sensing mechanism is based on the principle that gas analytes binding on the defects of the OLCs injects or extracts electrons from OLCs that disturb their conductance. Depending on the type of molecules adsorbed to the surface of defects in the C60 nanoparticles, specific effects of the p- or n-type semiconductors behavior manifests. The fast decrease in resistance of the OLC sensor under 10 ppm $H_2$ and $CH_4$ exposure can be attributed to the switch of the nanoparticles to n-type semiconductor behavior together with the additional increase of electrons number in the conduction band.

## 4. Conclusions

A room temperature working chemiresistive gas sensor was manufactured by dielectrophoresis with the purpose to detect hydrogen and methane compounds at the ppm level from human breath. Breath Testing is a useful, inexpensive, simple and safe diagnostic test in the diagnosis of carbohydrate maldigestion, methane-associated constipation, and evaluation of bloating/gas disorders. Onion-like carbons (OLCs), a part of the carbon fullerene C60 family, was used as sensing element based on their small, spherical, zero-dimensional (0-D) structures of 5 nm consisting of concentric shells of graphitic carbon. These nanoparticles manifest another advantage, based on the presence of synthesis-related defects in the OLC structure/layers that makes them suitable for developing CGSs without functionalization.

The manufactured sensors manifested good selectivity and good response time, with all the analyte molecules unbinding from the OLCs under 60 s. All sensors respond in the presence on both methane and hydrogen presence, and their functionality was proved under repeated exposures. Among all sensors, which were manufactured under different dielectroforetic voltages and frequencies, one of the sensors manifested enhanced selectivity towards methane, but the recovery time is prolonged to 200 s.

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## UTILIZAREA FULERENELOR C60 CA DETECTOR DE METAN ŞI HIDROGEN ÎN TESTELE DE RESPIRAȚIE

(Rezumat)

Testele de respirație bazate pe măsurarea cantității de hidrogen şi metan au fost utilizate cu succes pentru detectarea tulburărilor gastro-intestinale, pentru diagnosticarea sindroamelor de maldigestie al carbohidraților şi a supraaglomerării bacteriene în intestinul subțire (SIBO). Testul respirator (BT) este un test util, ieftin, simplu şi sigur în diagnosticul maldigestiei carbohidraților, al constipației şi al evaluării tulburărilor de balonare. Nanomaterialele pe bază de carbon reprezintă o soluție foarte promițătoare pentru proiectarea de noi senzori capabili să detecteze compuşii din respirație, iar recent, s-a observat o creştere constantă a eforturilor de cercetare şi a lucrărilor publicate privind utilizarea unor astfel de materiale ca element sensibil în fabricarea senzorilor. Nanoparticulele de carbon de tip fulerenă C60 cu multiple straturi concentrice, un membru al familiei de nanostructuri de carbon cu aplicații foarte promițătore, a făcut obiectul unei cercetări extinse de la descoperirea sa, datorită

prezenței defectelor structurale ce apar prin modul de sintezare al acestora. Aceasta este o caracteristică extrem de necesară în dezvoltarea dispozitivelor de detectare a gazelor ce lucrează la temperatură ambientală fără o sursa de încălzire externă. În această lucrare prezentăm un senzor de gaz chemirezistiv (CGS) bazat pe fulerene C60 care funcționează la temperatură ambientală, prezentând o sensibilitate sporită la $H_2$ şi $CH_4$.